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Valorisation of romanian peat for the removal of some heavy metals from aqueous media

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ABSTRACT

In this study, the sorption of some potential toxic heavy metal ions (Pb(II), Hg(II) and Cd(II), respectively) onto Romanian peat was investigated as a function of initial solution pH, sorbent dose, initial metal ions concentration and contact time, at room temperature (20 ± 0.5 ℃), in batch system. The uptake capacity of tested heavy metals decreased by decreasing the initial solution pH, suggesting that competition exists between hydrogen ions, present in high concentration in strong acid media and metal ions. The experimental data were evaluated using Langmuir and Freundlich isotherm models. The isotherm data followed the Langmuir model, and the maximum sorption capacity increases in the order Cd(II) < Pb(II) < Hg(II). The kinetics of metal ions sorption was rather rapid, with at least 68% of sorption occurring in 30 min. The experimental data were satisfactory correlated with pseudo-second-order kinetic model, which means that in the sorption mechanism the chemical interaction between positively charged metal ions and negatively charged functional groups from peat surface is the rate-controlling step. The results presented in this study showed that the Romanian peat could be used as a potential cost-effective sorbent for the treatment of complex aqueous media containing heavy metals.

Keywords: Sorption; Heavy metals; Romanian peat; Isotherm; Kinetics

1. Introduction

Nowadays, one of the most important environmental problems is the pollution caused by heavy metals. The main source of heavy metal pollution of aqueous environmental is determined mostly by human activities, such as processing of metal ores, mining, plating of metals, inadequate treatment of domestic and industrial wastewaters, etc. [1,2]. Due to their toxic effect, persistency, mobility and accumulation tendency, the removal of heavy metals has become one of the important issues in many industrialized countries, with respect to the environment and economical considerations. Among heavy metals, cadmium, lead and mercury have high priority for the removal from aqueous media [2,3], because they are considered the most potentially toxic heavy metals for environment.

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Methods for minimizing the concentration of heavy metal ions in surface waters and wastewaters are therefore crucial for environmental protection. The main techniques that have been used to remove heavy metal ions from aqueous media include chemical precipitation, membrane filtration, ion exchange and adsorption on activated carbon [4–8]. However, these methods are expensive or have some disadvantages such as are expensive, not eco-friendly and inefficient, in special when the heavy metals are present in low concentrations (1–100 mg dissolved metal/l), and may also generate secondary wastes, which are difficult to treat [8].

Compared with other conventional techniques used for metal ions removal, the sorption process has emerged as one of the economical and efficient method for the removal of heavy metal ions from aqueous media because of eco-friendly characteristics, low-cost, high uptake performances, less sludge production and possible regeneration of sorbents [9,10].

In the last years, considerable attention has been given to the use of natural materials, as sorbents, for the removal of heavy metals from contaminated aqueous media. This is due to the fact that the natural materials have high number and variety of superficial functional groups (such as carboxyl, carbonyl, hydroxyl, amino, phosphate, etc.), which can retain the hazardous substances through ion-exchange, complexation and adsorption. Moreover, they can be cheaply and easily obtained in considerable quantities [11]. One example of such material is peat.

The peat is a relatively inexpensive and widely available material in Romania and in many regions of the World, which is known to have a good sorption capacity for numerous metal ions [12,13]. Generally, the peat is considered a rather complex material with organic matter in various decomposition stages, which contains lignin, cellulose and humic substances as major constituents [11,14]. These constituents have numerous functional groups (such as hydroxyl, carboxyl, amino, carbonyl, etc.) that can be involved in chemical interactions with metal ions, as well as also responsible for the cation-exchange properties of peat. In some previous studies, we have show that the Romanian moss peat, sampled from surface (less than 0.5 m), is an effective sorbent, which can be used for the removal of several heavy metal ions from aqueous solution [15–17].

In this study, the same Romanian peat, sampled from a higher depth (1.5 m) was used as sorbent for the removal of Pb(II), Hg(II) and Cd(II) ions from aqueous media, in batch system. This material has a high decomposition degree, and the tests performed have shown that it has a low caloric power, and therefore, its use as a combustible is not profitable. In addition, the IR spectra recorded for this material have show that this peat has more carboxyl groups than the peat moss used previously, so it is expected to have better sorption characteristics for heavy metal ions from aqueous solution.

The sorption of considered heavy metals was examined by changing various experimental parameters (such as initial solution pH, sorbent dose, initial heavy metal concentration and contact time), at room temperature (20 ± 0.5°C). The equilibrium and kinetics of single-component sorption process were evaluated. Two isotherm models, Langmuir and Freundlich, have been used to determine the best-fitting model for the experimental data. The kinetics parameters of the sorption process for each heavy metal ion have also been examined and found that the pseudo-second-order kinetic model was applicable to all studied heavy metals, over entire time range.

2. Experimental

2.1. Materials

The peat used for the sorption experiments was drawing at 1.5 m depth from Poiana Stampei, Romania. It is of high decomposition degree, with the following characteristics: pH = 4.79 (in water), ash = 7.12%, total organic carbon = 32.17%, total oxygen = 13.75%, total nitrogen = 0.98%, total phosphorus = 3.01%. The peat samples was dried in air for 6 h at 70 ± 2°C, grounded and sieved until the particle dimension was less than 1 and 2 mm and then stored in desiccators for the further use.

The stock solutions of heavy metals ions (Pb(II), Hg(II) and Cd(II), respectively) contain around 10^{-2} mol M(II)/l were prepared by metal nitrate (from Reactivul Bucharest) dissolving in double-distilled water, followed by solutions standardization [18]. The working solutions were obtained by diluting the stock solutions with double-distilled water. The values of initial solution pH were adjusted with 0.1 mol/l HNO_3 or NaOH solutions. All chemical reagents were of analytical degree and were used without further purifications.

2.2. Sorption experiments

The sorption experiments were performed for a single component, at different initial solution pH, peat dosage, heavy metal ions concentration and contact time, at room temperature (20 ± 0.5°C), in 150 ml conical flasks with intermittent stirring (usually 24 h). The influence of initial solution pH on the sorption capacity of Pb(II), Hg(II) and Cd(II) onto Romanian peat was examined by mixing 0.125 g of dried peat with 25 ml of 0.35 mmol M(II)/l solution, and adjusting the pH value between 2.0 and 6.0 (measured with a Radelkis OK-281 pH/ion-meter, equipped with a combined glass electrode). The effect of Romanian peat dosage on heavy metals sorption was also determined by mixing sorbent samples (between 3.0 and 40 g/l) with 25 ml of heavy metal ions solution (0.35 mmol/l) and pH = 5.5, at room temperature. The influence of heavy metal ions concentration on Romanian peat sorption capacity was studied between 0.2 and 2.0 mmol/l concentration range. For kinetic experiments, a constant Romanian peat dose of 5 g/l was mixed with 25 ml of 0.8 mmol M(II)/l metal ions solution (Pb(II), Hg(II) and Cd(II), respectively), at various time intervals between 5 and 180 min. After the sorption procedure was complete, the phases were separated by filtration and the heavy metals concentrations in filtrate have been determined spectrophotometrically (Digital Spectrophotometer S 104 D, 1 cm glass cell) [19,20], using a prepared calibration graph. All the experiments were carried out in duplicates with the average presented in the results.

The efficiency of the sorption process of considered heavy metals (Pb(II), Hg(II) and Cd(II)) onto Romanian peat was quantitatively evaluated using amount of heavy metals retained on weight unit of peat (q, mmol/g) and the percent of heavy metals removal (R,%). These parameters were calculated from experimental results with a relative standard deviation less than ± 0.5%, using the following relations:

$$q = \frac{(c_0 - c) \times (V/1000)}{m} \tag{1}$$

$$R = \frac{c_0 - c}{c_0} \times 100 \tag{2}$$

where c_0 is the initial concentration of heavy metal ions in aqueous solution (mmol/l), c is the concentration of heavy metal ions at equilibrium (mmol/l), V is the volume of aqueous solution (l), and m is the mass of dry peat (g).

The Romanian peat samples obtained after filtration were dried in air (4 h, at 70–75°C), mortared and then used for the FT-IR spectra recording (Bio-Rad FT-IR Spectrometer, 400–4,000 cm^{-1} spectral domain, KBr pellet method).

3. Results and discussion

3.1. Effect of initial solution pH

The pH of aqueous solution is one of the most important parameter controlling the uptake capacity of heavy metals from aqueous media, because its value affects both the solubility and speciation of metal ions, and the dissociation degree of functional groups from sorbent surface [21]. In this study, the sorption of considered heavy metals (Pb(II), Hg(II) and Cd(II), respectively) was examined in the initial solution pH range between 2.0 and 6.0, and the obtained results are illustrated in Fig. 1. According to the speciation diagrams, in the studied pH and, the Pb(II) and Cd(II) ions are in aqueous solution predominantly as free ions (M^{2+}), while Hg(II) exist as $HgOH^+$ at pH values higher than 4 [22,23].

It can be observed from Fig. 1 that the sorption efficiency gradually increased with the increase in initial solution pH, to attain a maximum at pH 5.5, for all studied heavy metals. After this value, a slow decrease in sorption efficiency was observed, especially in case of Pb(II) and Cd(II) ions, and this is probably due to the start of the precipitation of these cations. At low pH values, the decrease in sorption efficiency could be explained by an increase in competition between protons and metal ions for binding sites of peat. In this case, the peat surface was more positively charged and this makes that the efficiency of sorption process to decrease. The increasing in the initial solution pH determined the increase of dissociation degree of functional groups from sorbent surface, and in consequence, the number of electrostatic interactions between negatively charged sorbent and positively charged metal ions will increase.

A rather different behaviour is observed in case of Hg(II) sorption, where even at low initial solution pH, the obtained values of removal percent are relatively high. This is due to the fact that Hg(II) species [23] can be easily bonded to donor atoms of functional

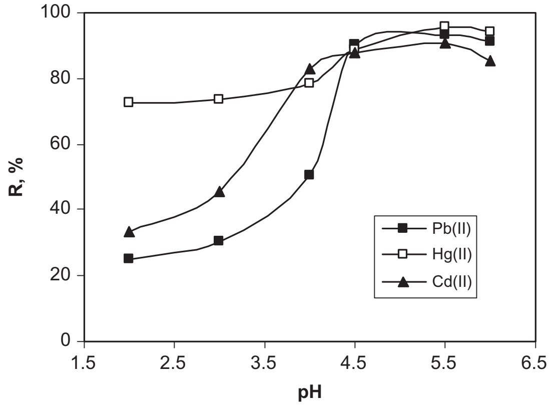

Fig. 1. Effect of initial solution pH on Pb(II), Hg(II) and Cd (II), respectively, removal by sorption onto Romanian peat (c_0 = 0.35 mmol/l, sorbent dose = 5.0 g/l, time = 24 h, temperature = 20°C).

groups from sorbent surface by other kind of interactions, than electrostatic, and the influence of initial solution pH is less pronounced than in case of Pb(II) and Cd(II) sorption.

Starting from these observations, an initial solution pH of 5.5 was considered as optimum for the sorption of considered heavy metal ions (Pb(II), Hg(II) and Cd (II), respectively) onto Romanian peat, and this value was used for the further experiments.

3.2. Effect of adsorbent dosage

The effect of Romanian peat dose on the sorption of Pb(II), Hg(II) and Cd(II) ions was studied by changing the sorbent dose from 3.0 to 40.0 g/l, employing an initial heavy metal ions concentration of 0.35 mmol/l and an initial solution pH of 5.5 (Fig. 2).

The obtained experimental results showed that the sorption efficiency is not significant influenced by the increase in sorbent dose. Thus, by varying the sorbent dose from 3.0 to 40.0 g/l, a slow increase in the removal percents was obtained for all studied heavy metals (from 95.15 to 99.71% in case of Pb(II), from 92.54 to 99.32% in case of Hg(II), and from 89.13 to 99.65% in case of Cd(II), respectively). Further increase in the sorbent dosage does not change significantly the values of removal percents. The observed behaviour can be attributed to the increase in the positive binding sites number as the sorbent dosage rises and then reaching a saturation point after which no further heavy metals sorption occurs [24]. Therefore, the optimum sorbent dose for the removal of considered heavy metal ions (Pb(II), Hg(II) and Cd(II)) was selected as 5.0 g/l for further experiments, because for this value of sorbent dose the values of removal percents (*R*,%) are higher than 90% for all studied heavy metal ions.

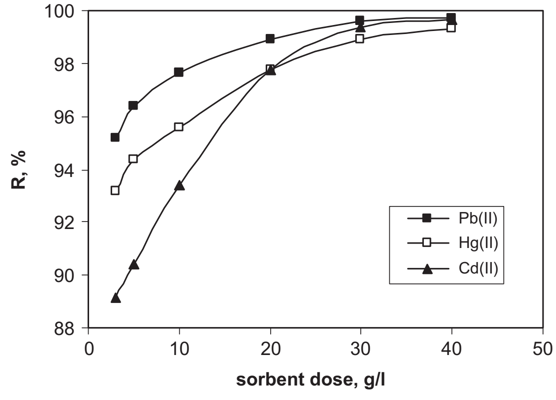

Fig. 2. Effect of Romanian peat dosage on Pb(II), Hg(II) and Cd(II) sorption ($c_0 = 0.35$ mmol/l, pH = 5.5, time = 24 h, temperature = 20°C).

3.3. Effect of initial heavy metal ions concentration

For all studied heavy metals (Pb(II), Hg(II) and Cd (II)), a significant influence of the initial heavy metal ions concentration on the sorption process onto Romanian peat was observed. In this study, the initial concentration of heavy metals was varied from 0.2 to 2.0 mmol/l, while the sorbent dose and initial solution pH were maintained at constant values establish in the previous experiments. Fig. 3 illustrates the effect of initial concentration of heavy metal ions on their removal percent (a) and of sorption capacity (b).

The values of removal percent (*R*,%) decrease from 95.13 to 55.3% for Pb(II), from 95.54 to 72.12% for Hg (II) and from 94.43 to 40.95% for Cd(II), respectively, when the initial heavy metals concentration increase from 0.2 to 2.0 mmol/l, in mentioned experimental conditions. On the other hand, the sorption capacity of Romanian peat (*q*, mmol/g) increases with the increase in the initial heavy metal ions concentration. This is a typical behaviour, which has been reported in many studies from the literature [25–27], and is due to the fact that an increase of initial metal ions concentration provides a higher driving force to overcome all mass transfer resistances between sorbent and aqueous solution. However, at high initial heavy metal ions concentrations, the available superficial functional groups are already occupied, and in consequence, the diffusion of heavy metal ions to un-react functional groups is inhibited.

The differences observed in the sorption behaviour of the three considered heavy metals onto Romanian peat are probably, determined by their different affinity for the functional groups from sorbent surface. This hypothesis is confirmed by the FT-IR spectra recorded for dry sorbent before and after the retention of Pb(II), Hg(II) and Cd(II) ions from aqueous solution (Fig. 4).

The comparison of obtained spectra indicate that after each heavy metal sorption, the band from 1,730 cm^{-1}, corresponding to carbonyl stretching of un-dissociated carboxyl groups of adsorbent is attenuated (in case of Cd(II) and Pb(II) sorption) or even disappear (in case of Hg(II) sorption). In addition, the band from 1,099 cm^{-1}, is due to C–O stretching of carbonyl groups and the bending vibration of hydroxyl groups from spectra (1), but the attenuation or disappearance of this band after heavy metals sorption (spectra 2, 3, 4), suggest that these types of functional groups are likely to participate in heavy metals binding.

The changes observed in the FT-IR spectra indicate that the sorption processes of studied heavy metals (Pb(II), Hg(II) and Cd(II), respectively) from aqueous

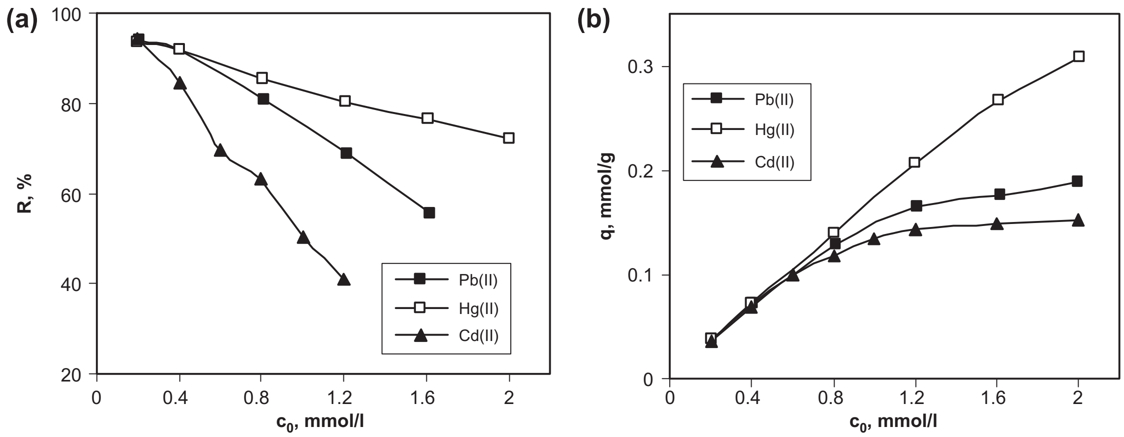

Fig. 3. Effect of initial heavy metals concentration on the sorption onto Romanian peat: (a) removal percent; (b) sorption capacity (pH = 5.5, sorbent dose = 5.0 g/l, time = 24 h, temperature = 20°C).

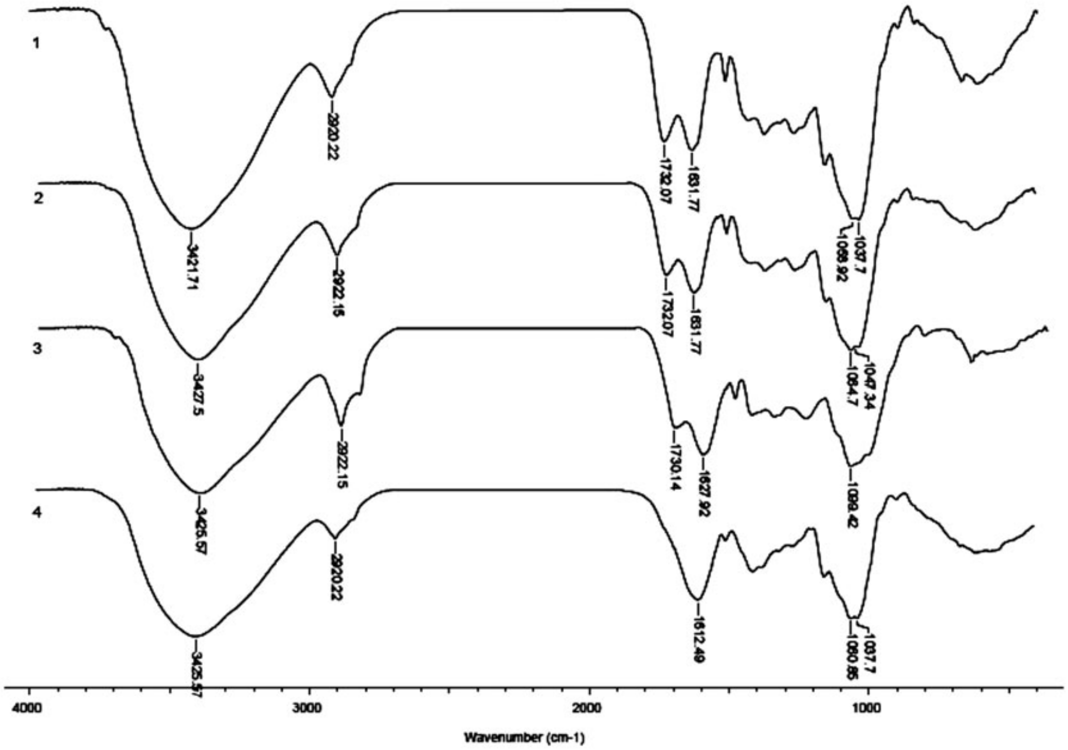

Fig. 4. FT-IR spectra of dry Romanian peat before (1) and after heavy metals sorption: Cd(II)—(2), Pb(II)—(3) and Hg (II)—(4).

solutions onto Romanian peat occurs predominantly by electrostatic interactions.

3.4. Effect of equilibrium contact time

Fig. 5 shows the effect of contact time for the sorption of considered heavy metals (Pb(II), Hg(II) and Cd (II)) onto Romanian peat.

The experimental results indicate that the amount of each heavy metal retained on Romanian peat increases rapidly with the contact time, up to 30 min for Pb(II), 45 min for Hg(II) and 90 min for Cd(II), respectively. After this faster step, the sorption rate becomes slower, and finally no further significant sorption is noted beyond 3 h. The different values of contact time necessary for to reach the maximum of

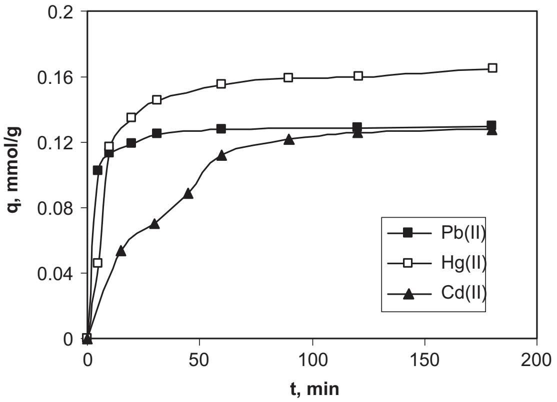

Fig. 5. Effect of contact time for Pb(II), Hg(II) and Cd(II) sorption onto Romanian peat ($c_0 = 0.80$ mmol/l, pH = 5.5, adsorbent dose = 5.0 g/l, temperature = 20˚C).

sorption are another argument which sustain the different affinity of the considered heavy metals towards the functional groups from sorbent surface. In addition, the relatively fast sorption of Romanian peat makes this material suitable for continuous flow water treatment system.

3.5. Sorption isotherm

In this study, the experimental results were fitted using two linear isotherm models, Langmuir and Freundlich, and the best fit isotherm model was selected based on the linear regression coefficients (R^2). The mathematical expressions of this two isotherm models are [28,29]:

Langmuir isotherm model

$$\frac{1}{q} = \frac{1}{q_{\max}} + \frac{1}{q_{\max} \times K_L \times c} \tag{3}$$

Freundlich isotherm model

$$\lg q = \lg K_F + \frac{1}{n} \lg c \tag{4}$$

where $q_{\max}$ is maximum sorption capacity upon complete saturation of sorbent surface (mmol/g); K_L is the Langmuir constant, related to the sorption/desorption energy (l/mmol); K_F is the Freundlich constant and represent an indicator of the sorption capacity and n is a constant that characterized the surface heterogeneity.

The graphical representation of the liniarized forms of Langmuir and Freundlich isotherm models for the sorption of Pb(II), Hg(II) and Cd(II), respectively, onto Romanian peat, at room temperature (20 ± 0.5˚C) is presented in Fig. 6, and the isotherm parameters, calculated on the basis of the models equations, are summarized in Table 1.

The values of correlation coefficients (R^2) indicate that the sorption isotherm data of studied heavy metals (Pb(II), Hg(II) and Cd(II), respectively) are very well represented by the Langmuir model and confirm the monolayer sorption of these heavy metal ions from aqueous media onto sorbent surface.

The maximum sorption capacity ($q_{\max}$, mmol/g), which is a measure of the sorption capacity to form a monolayer, calculated from Langmuir isotherm equation, increased in the order: Cd(II) < Pb(II) < Hg(II). The high values of $q_{\max}$ obtained in case of Hg(II) ions shows that this heavy metal ion has the higher affinity for the functional groups of sorbent, and can be most easily retained. In addition, the Langmuir constant values (K_L), which denotes the sorption energy, are high values in all cases, suggesting that between functional groups from sorbent surface and the heavy metal ions, strong interactions (mainly electrostatic) occur.

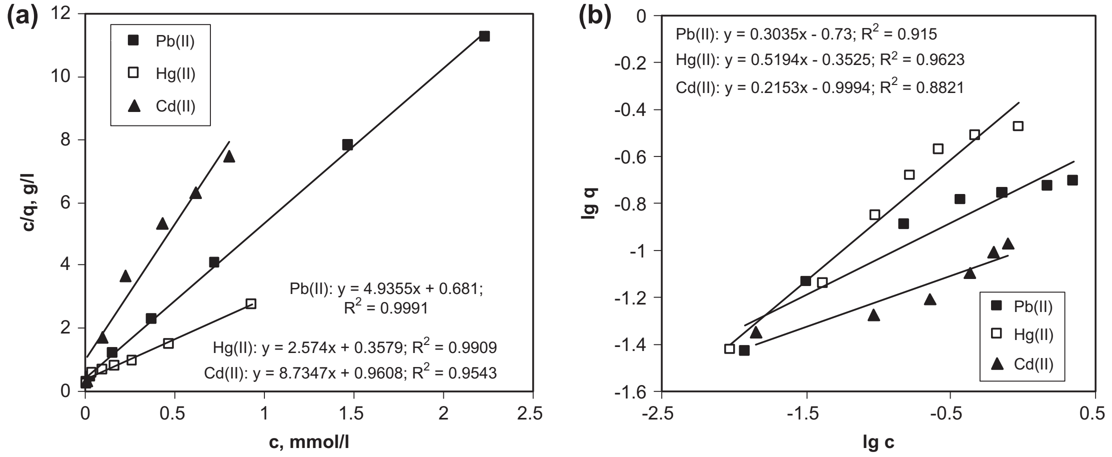

Fig. 6. Langmuir (a) and Freundlich (b) representation for the sorption of Pb(II), Hg(II) and Cd(II) ions from aqueous media onto Romanian peat.

Table 1
Isotherm parameters for considered heavy metals sorption by Romanian peat

Heavy metal	Langmuir isotherm model				Freundlich isotherm model		
	R^2	$q_{\max}$, mmol/g	K_L, l/g	R_L	R^2	n	K_F, mmol $l^{1/n}$/g mmol$^{1/n}$
Pb(II)	0.9909	0.2026	7.2479	0.9816	0.9623	1.9253	0.4441
Hg(II)	0.9991	0.3885	7.1919	0.9771	0.9150	3.2949	0.1862
Cd(II)	0.9543	0.1145	9.0899	0.9892	0.8821	4.6446	0.1000

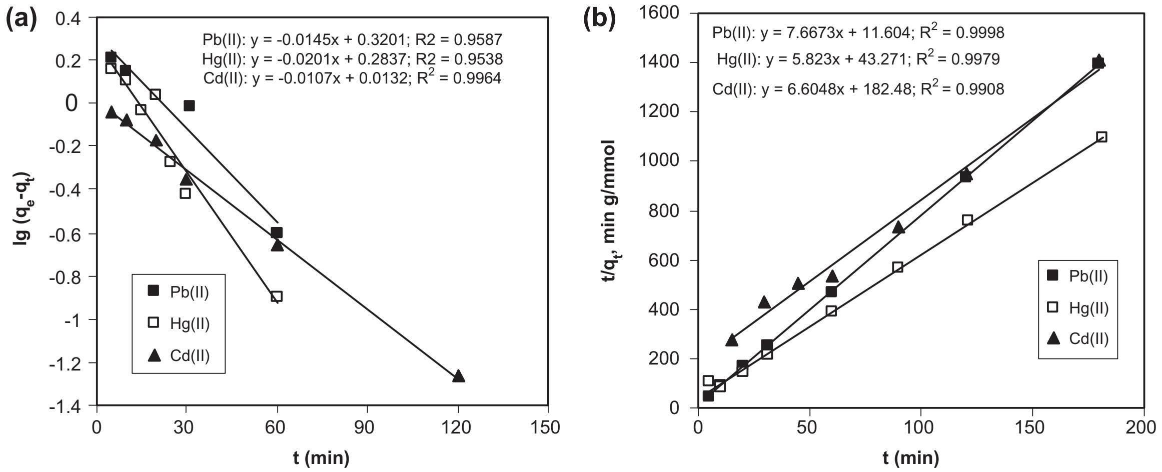

Fig. 7. Pseudo-first-order (a) and pseudo-second-order (b) kinetics of heavy metal ions (Pb(II), Hg(II) and Cd(II), respectively) sorption onto Romanian peat.

The Langmuir isotherm can also be expressed by a dimensionless constant (R_L), which is referred as a separation factor or equilibrium parameter [30]. This constant provides information about whether the sorption process is spontaneous or non-spontaneous and is calculated by using the following relation:

$$R_L = \frac{1}{1 + K_L c_0} \tag{5}$$

where c_0 is the highest initial heavy metal ions concentration (mol/l).

When the R_L values lie between 0 and 1, the adsorption process is favourable [30]. The values of R_L calculated from Eq. (5) for all three studied heavy metals (Pb(II), Hg(II) and Cd(II), respectively) are also presented in Table 1. It can be observed that the R_L values obtained in case sorption of considered heavy metal ions onto Romanian peat are between 0.97 and 0.99, and in consequence, the sorption process can be considered a spontaneous one, in each case.

3.6. Sorption kinetics

In order to investigate the mechanism of considered heavy metals sorption onto Romanian peat, the pseudo-first-order and pseudo-second-order kinetics models were tested to fit the kinetics experimental data. The mathematical equations of these two models are expressed by the following relations [31,32]:

pseudo-first-order kinetic model

$$\lg\,(q_e - q_t) = \lg\,q_e - k_1 \times t \tag{6}$$

pseudo-second-order kinetic model

$$\frac{1}{q_t} = \frac{1}{k_2 \times q_e^2} + \frac{t}{q_e} \tag{7}$$

where q_e is the sorption capacity at equilibrium (mmol/g); q_t is the sorption capacity at time t (mmol/g); t is the sorption time (min); k_1 is the rate constant of pseudo-first-order kinetic model (min^{-1}) and k_2 of the rate constant of the pseudo-second-order kinetic model (g/mmol min).

Table 2
The kinetics parameters obtained for considered heavy metals sorption onto Romanian peat

Heavy metal	q_e^{exp}, mmol/g	Pseudo-first-order model			Pseudo-second-order model		
		R^2	q_e, mmol/g	k_1, min^{-1}	R^2	q_e, mmol/g	k_2, g/mmol min
Pb(II)	0.1292	0.9587	2.0897	0.0145	0.9998	0.1304	5.0664
Hg(II)	0.1705	0.9538	1.9217	0.0201	0.9979	0.1717	0.7836
Cd(II)	0.1529	0.9964	1.0308	0.0107	0.9908	0.1514	0.2395

The linear representations of the pseudo-first-and pseudo-second-order kinetics models for the sorption of considered heavy metal ions (Pb(II), Hg(II) and Cd (II), respectively) onto Romanian peat are illustrated in Fig. 7, and the values of kinetic parameters obtained in each case and the corresponding correlation coefficients (R^2) are summarized in Table 2.

It is obvious that the correlation coefficients ($R^2 > 0.99$) for the pseudo-second-order kinetic model are higher in comparison with the pseudo-first-order kinetic model, and the calculated values q_e, mmol/g for the pseudo-second order kinetic model are very close to the experimental values (q_e^{exp}, mmol/g) for all studied heavy metal ions. These observations indicate that the pseudo-second-order kinetic model was better in describing the sorption kinetics of considered heavy metals onto Romanian peat. The pseudo-second-order kinetic model is based on the assumption that the rate-controlling step in the sorption process are the chemical interactions between superficial functional groups of sorbent and considered heavy metal ions, and similar behaviour have been reported for various types of low-cost materials [33,34].

In addition, the pseudo-second-order rate constant increase in the order: Cd(II) ($k_2 = 0.2395$ g/mmol min) < Hg(II) ($k_2 = 0.7836$ g/mmol min) < Pb(II) ($k_2 = 5.0664$ g/mmol min), suggesting that the chemical interactions depends by the affinity of heavy metal ion to interact with the superficial functional groups of sorbent.

4. Conclusions

The results presented in this study indicate that the Romanian peat, sampled from a relatively high depth (1.5 m) can be successfully used for the removal of some potential toxic heavy metals (Pb(II), Hg(II) and Cd(II), respectively). The experiments were performed in batch systems, as a function of initial solution pH, sorbent dose, initial heavy metal ions concentration and contact time, at room temperature (20 ± 0.5 °C). The obtained results have shown that the maximum amounts of heavy metals retained were achieved at pH 5.5 and 5 g sorbent/l.

The Langmuir and Freundlich isotherm models were used to provide a mathematical description of the data for the sorption process of considered heavy metals from aqueous solution. The obtained results indicate that the sorption equilibrium data were very well fitted by the Langmuir isotherm model in all cases, and the maximum sorption capacity increase in the order: Cd(II) ($q_{\max} = 0.1145$ mmol/g) < Pb(II) ($q_{\max} = 0.2026$ mmol/g) < Hg(II) ($q_{\max} = 0.3885$ mmol/g), under employed experimental conditions.

The pseudo-second-order model best describes the sorption kinetics of the studied heavy metals (Pb(II), Hg(II) and Cd(II), respectively) onto Romanian peat, over entire time range. The obtained values confirm that the chemical interactions play an important role in the sorption mechanism, and this hypothesis was also sustained by the FT-IR spectra data.

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